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Technical Translation 142

COMPRESSIBILITY OF NITROGEN-HYDROGEN-  
AMMONIA MIXTURES AT HIGH  
PRESSURES AND TEMPERATURES

by

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November 1968

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Dover, N.J.

## COMPRESSIBILITY OF NITROGEN-HYDROGEN-AMMONIA MIXTURES AT HIGH PRESSURES AND TEMPERATURES

Ya. S. Kazarnovskiy, G. B. Simonov and G. Ye. Aristov,  
Zhurnal Fizicheskoy Khimii (Journal of Physical  
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Data available in scientific literature on the compressibility of gas mixtures deal primarily with mixtures consisting of non-dipole gases. The mixtures of nitrogen-hydrogen (1), hydrogen-carbon dioxide (2), nitrogen-carbon dioxide (2), nitrogen-methane (3), hydrogen-methane (3), and methane-ethane (4) have been investigated in a broad range of temperatures, pressures and compositions.

Data on the compressibility of gas mixtures, one component of which is a dipole gas, however, is completely unsystematic (5), and is available only up to a pressure of 100 atmospheres.

In our experiment we have determined the compressibility of binary ammonia-hydrogen and ammonia-nitrogen mixtures, as well as of ternary mixtures containing nitrogen, hydrogen and ammonia, for a number of compositions at high temperatures and pressures.

### Experimental Part.

For determining the compressibility of nitrogen-hydrogen-ammonia mixtures, we chose the Michels (6) method, who used it to obtain exceptionally great precision. The diagram of the Michels method is shown on Fig. 1.

The piezometer d consists of a glass vessel formed of several consecutively joined bulbs. The capillaries between the bulbs contain platinum wires fused into the glass, with their ends externally connected to a common platinum spiral e. The wide end of the piezometer enters into a metal container f containing mercury h. The millivoltmeter g measures the potential difference at the ends of the platinum spiral e. The volume of the parts of the piezometer between the Platinum wires fused in and the upper capillary section has been accurately calibrated. If pressure is exerted by the oil upon the mercury, then the latter will rise inside the piezometer, and consecutively close

the platinum wire contacts. The instant of contact closing is registered by a jump of the millivoltmeter needle; the pressure and temperature are noted at the same time. Thus, for the given system, several values of  $p - v$  can be obtained, corresponding to the number of platinum contacts within the piezometer. By varying the temperature and repeating the same operation it is possible to obtain several isotherms with one filling of the piezometer with gas.

Fig. 1. Diagram of the Michels method.

KEY: a. Battery

b. Resistances

c. Vessel

d. Piezometer

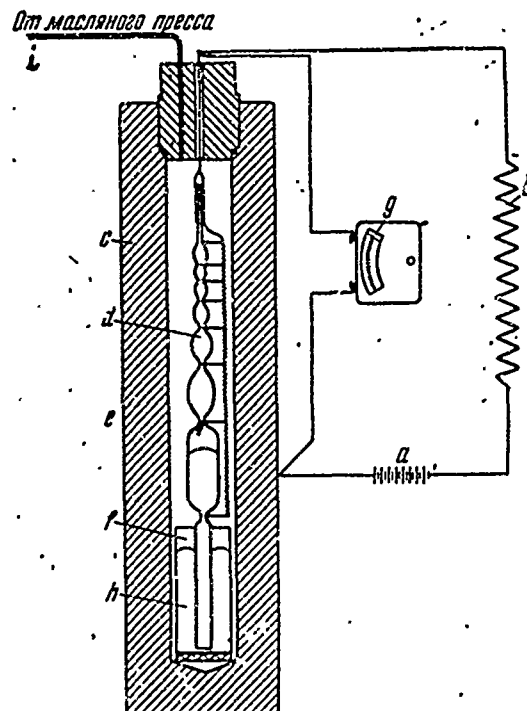
e. Platinum spiral

f. Metal container

g. Millivoltmeter

h. Mercury

i. Oil inlet under pressure



The design of this basic apparatus (Fig. 1) for determining the compressibility of nitrogen-hydrogen-ammonia mixtures at high pressures is somewhat different from Michels' apparatus.

The preparation of a mixture of gases, one of which is ammonia, under high pressure presents considerable difficulty for the experimenter.

The process of spontaneous mixing of gases under pressures of about 100 atmospheres requires several days. Since the critical temperature of ammonia is  $132.9^{\circ}\text{C}$ , the mixing must take place at a constant heating above  $132.9^{\circ}\text{C}$ . In order to avoid such long heating periods, the mixing of the gases was performed in a "mixer" autoclave, previously evacuated to a pressure of  $10^{-2}$  mm Hg and placed in a constant temperature chamber which was filled with oil of the "Frikus" type, heated to  $150^{\circ}\text{C}$ . The mixer could rotate along its horizontal axis, and contained a ball which mixed the gas by rolling from one end to another. Completion of the mixing process was determined by a number of congruent analyses of the gas mixture for ammonia, performed with a 0.03% accuracy.

The diagram of the apparatus (Fig. 2) which was used to determine the compressibility of nitrogen-hydrogen-ammonia mixtures is similar to the one used by one of us to study the compressibility of ammonia at high pressures (7).

The design of our apparatus is represented in Fig. 2.

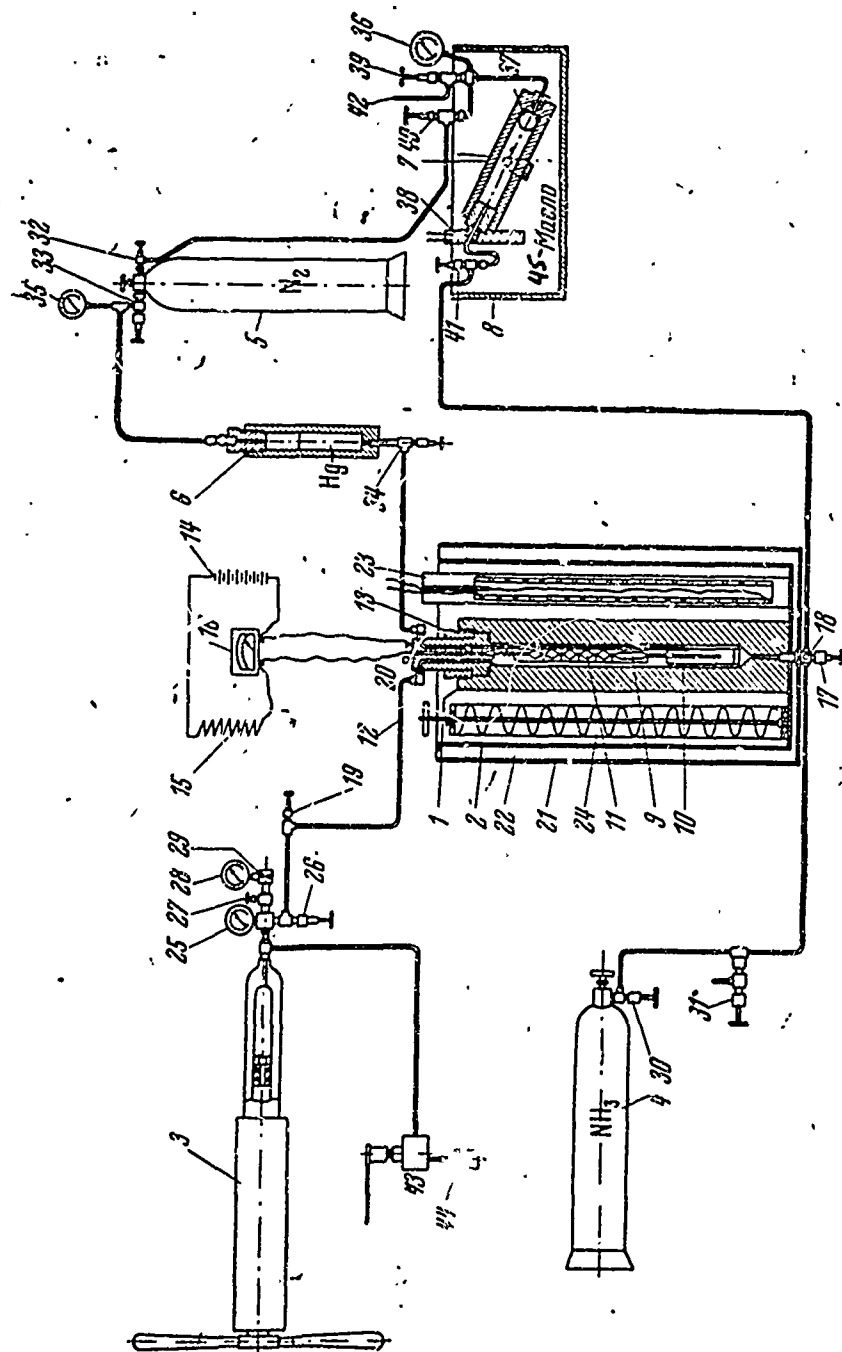


Fig. 2. Apparatus for determining the compressibility of nitrogen-hydrogen-ammonia mixtures.



KEY for Fig. 2.

- |   |  |
|---|--|
| 1. Autoclave                              | 19. Oil inlet valve                                |
| 2. Inner casing of the thermostat chamber | 20. Oil outlet valve                               |
| 3. Oil press                              | 21. Outer thermostat casing                        |
| 4. Ammonia tank                           | 22. Asbestos                                       |
| 5. Nitrogen tank                          | 23. Electric heater                                |
| 6. Mercury column                         | 24. Snake-type mixer                               |
| 7. Mixer                                  | 25. Manometer for 2000 kg/cm <sup>2</sup>          |
| 8. Mixer thermostat                       | 26-27. Hofer valves                                |
| 9. Piezometer                             | 28. Sampling manometer                             |
| 10. Mercury container                     | 29-34. Hofer valves                                |
| 11. Platinum spiral of the piezometer     | 35. Manometer for 200 kg/cm <sup>2</sup>           |
| 12. Autoclave pressure head               | 36. Manometer for 400 kg/cm <sup>2</sup>           |
| 13. Fastening nut                         | 37. Mixing ball                                    |
| 14. Battery                               | 38. Electric heater                                |
| 15. Resistance chamber                    | 39-41. Hofer valves                                |
| 16. Millivoltmeter                        | 42. Pipe for bleeding off gas samples for analysis |
| 17. Gas inlet valve                       | 43. Oil pump                                       |
| 18. Triple coupling                       | 44. Oil tank                                       |
|   | 45. Oil  |

The measurements were carried out as follows: first the three electric heaters were turned on (23), heating the "Vapor T" oil filling the thermostat (2).

The oil was intensively mixed with the three snake-type mixers (24). Then the autoclave (1) with the piezometer (9) and metal vessel (10) contained therein was evacuated for a 30 minute period at 150°C by using the Kamovskiy oil pump, down to a pressure of  $10^{-2}$  mm Hg. Then, the gas mixture prepared as described above, was transferred by pipes from the mixer to the autoclave, filling it up to the valves (19) and (34). The tubes carrying and filled by the mixture were electrically heated to about 200° in order to avoid possible condensation of ammonia out of the mixture. Then, mercury from the column (6) was transported by nitrogen pressure along the tube and into the metal container (10), blocking the gas mixture's exit from the piezometer, and preventing the nitrogen following it from entering the piezometer.

The gas mixture in the autoclave but outside the piezometer was released through valve (20) at the same time that the autoclave was filled with oil by using the oil press (3). Temperature was kept constant by using rheostats, within the limits of  $\pm 0.15^\circ\text{C}$ . According to Michels, approximately one hour is required for an equalization of the temperature inside the autoclave with the oil temperature in the thermostat. After this one hour period, the pressure of the gas mixture in the piezometer was raised by sending oil from the press (3) into the autoclave. The mercury being raised inside the piezometer by this gradually closed the platinum contacts one after the other. The pressure corresponding at a given temperature to a definite volume of gas in the piezometer was taken as an average of pressures shown by the manometers during

the making and breaking of contacts. These divergencies constituted a maximum of 0.1% of the measured pressure. Before taking the pressure reading when one or another contact was closed, we waited for 10-15 minutes, this period being necessary for an equalization of temperature which had changed within the autoclave as the result of compression of the gas mixture and addition of cold oil. Since the flash point of the "Vapor T" oil contained in the thermostat is about 315°C, the maximum temperature we reached was 300°C. The piezometer we used is shown in Fig. 3. It had seven platinum contacts fused in, and its total working volume was about 8 cm<sup>3</sup>. The gas was not compressed completely to the last, seventh contact, and it served for the purpose of calibration of the upper capillary. The diameter of the capillary between the contacts was 0.6-0.7 mm. The contacts were made of 0.3 mm diameter platinum wire. The platinum wire joining the contacts was wound in spiral form, had a diameter of 0.2 mm, and was insulated by glass capillary tubes. The piezometer was made of No. 23 glass (so-called Riting), possessing a volumetric expansion coefficient of  $260-270 \cdot 10^{-7}$ . At the maximum temperature of 300°C, the volume of correction for heat expansion of the glass was 0.064 cm<sup>3</sup> with a volume of 8 cm<sup>3</sup>.

The piezometer was calibrated by weighing the mercury flowing out of it, as it was breaking the contacts with the platinum. The moment of breaking contact was fixed by a jump of the millivoltmeter needle. The accuracy of calibration in relation to the largest volume was 0.014% and to the smallest - 0.07%. The mercury used in the experiment was purified in advance by distillation in vacuum.

The ammonia that was one of the components of the gas mixtures we studied contained up to 0.2% nitrogen and hydrogen and 0.07% water in its commercial form. We removed the nitrogen and hydrogen completely by letting one-third of the ammonia in the tank out into the air, after which the gas taken from the tank for analysis was completely absorbed by sulfuric acid. The water was not removed from the ammonia, since such a negligible content cannot have a noticeable effect on the values of data obtained. The other components of our mixtures, nitrogen and hydrogen, contained up to 0.2% oxygen in their commercial form. The complete removal of oxygen was accomplished by passing these gases over red copper heated to 500°C at a pressure of 120 atmospheres. In transferring the gas mixture from the mixer to the autoclave, no condensation of ammonia from the mixture took place: analyses for ammonia of the gas mixture from the mixer and taken directly from the autoclave always coincided excellently.

The temperature was measured by a set of ordinary Anschütz thermometers with 0.2° divisions. In order to avoid the necessity of introducing a correction for the protruding mercury bulb, the thermometer was placed in a special periscope (Fig. 4) in which it was located below the oil level in the thermostat. The periscope was made of red copper sheet, and the mercury bulb of the thermometer was inside a closed tube also of red copper sheet of 0.1 mm thickness. This tube was filled with Wood's alloy for better heat transfer. The lamp was lit up only at the moment of taking a temperature reading, and had no effect on the thermometer reading. We shall describe our taking of pressure measurements in somewhat greater detail.

At the present time, piston manometers are used for measuring pressure in measurements involving high pressures, particularly in determining the compressibility of gases. This is due to the fact that the accuracy of ordinary spring-type, so-called "burdonovskiye" manometers is about  $1\frac{1}{2}$ -2 $\frac{1}{2}$ % of the measured values. The indicated error is to a significant degree caused by the fact that in use, the spring-type manometer is under load for long periods of time which is one of the

Fig. 3.

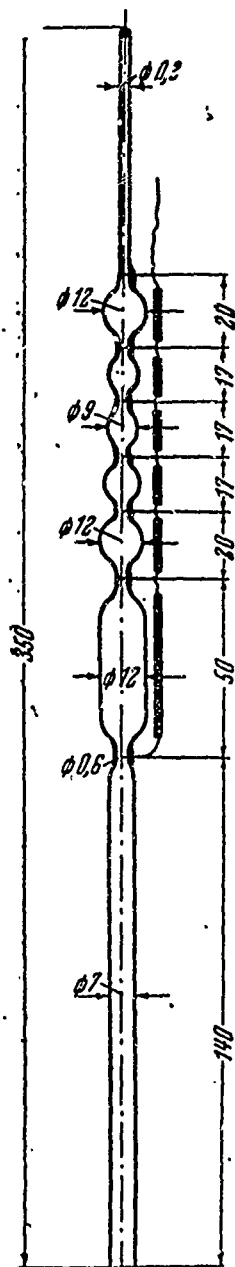
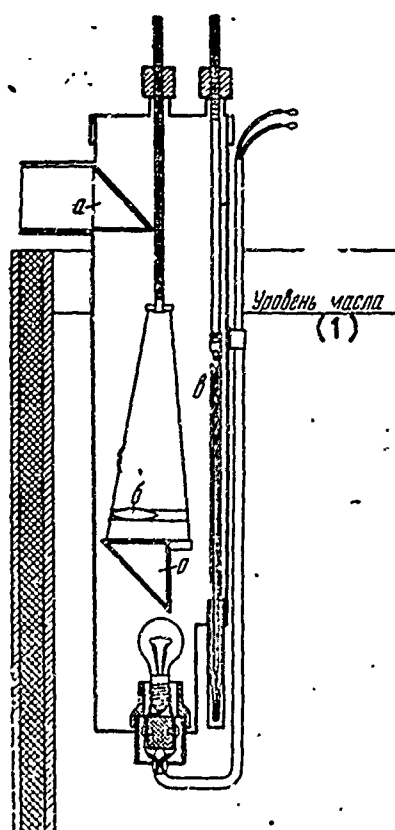


Fig. 4.  
The periscope



KEY: a Prism

b Thermometer

6 Magnifying glass

1) Oil level

main causes of residual deformations appearing in them. In 1937, the State Institute of Metrology developed spring-type, so-called "model manometers," with a 300° scale. Discrepancies in the readings of these manometers with direct and reverse operation are 0.3°. Their accuracy, according to the guarantee of the Institute of Metrology is 0.35% of the measured pressure.

Measurement of pressures in our experiment were carried out on three "model manometers" for 500, 1000 and 2000 kg/cm<sup>2</sup>.

These manometers were contained in pipes filled with oil, and were turned on only at the moment of closing of the platinum circuit in the piezometer, after a preliminary time interval of 10-15 minutes which was necessary for dissipation of the heat of compression of the gas. Preliminary measurement of pressure when the circuit was completed was accomplished by an ordinary spring-type manometer. Thus, the "model manometers" were under load for 3-4 minutes, i.e., they were exposed to the same conditions by us as they are at the Metrology Institute when used for the calibration of other manometers.

#### Experimental Results.

Since in the Michels method, the quantity of gas taken for the determination of its compressibility is not measured, we calculated the molar volume values of the nitrogen-hydrogen-ammonia mixtures for the pressures of 90-128 atmospheres by the Beattie-Bridgeman equation (8). This equation was verified by Beattie (9) and Beattie-Ikehara (10) for pressures up to 300 atmospheres for a large number of gas mixtures, reproducing the experimental data of various authors with an average accuracy of 0.5%. The degree of accuracy of this equation for nitrogen-hydrogen-ammonia mixtures was tested by us by the following method.

For a gas mixture of a certain composition, four Beattie-Bridgeman equations were set up for the temperatures of 150, 200, 250 and 300°C. For the equation compiled at the temperature of 250°C, at which the polar nature of the ammonia molecule will be little evident, a value is selected for the volume, corresponding to the experimentally obtained pressure at the first contact of the piezometer. Further, this value of volume is inserted into the other three equations, and the calculated pressures are compared with experimentally determined values. At the temperatures of 300 and 200°C,  $p$  calculated always coincided well with  $p$  experimental. At 150°C, divergencies of up to 2 atmospheres were observed in some experiments. This, of course, could be explained by the fact that at 150°C the mixture approaches the critical temperature of ammonia, near which the polar nature of its molecule will be especially evident.

For ammonia we used the values of the Beattie-Bridgeman equation constant as determined by Beattie and Lourrence (11), and for hydrogen and nitrogen, those compiled by Deming and Shupe (12) for less than critical densities. Table 1 shows the four Beattie-Bridgeman equations we compiled for the ammonia-hydrogen mixture containing 34.75% ammonia by volume, as well as our values of  $p$  calculated and  $p$  experimental.

Table 1. The Beattie-Bridgeman equation for the ammonia-hydrogen gas mixture, containing 34.75% ammonia. (1) - calculated pressure (2) - experimentally determined pressure.

$t^{\circ}\text{C}$	Уравнение состояния Битти-Бриджмена	$P_{\text{расч}}, \text{ атм}$	$P_{\text{эксп}}, \text{ атм}$
150	$p = \frac{34,730}{v} - \frac{0,4963}{v^2} - \frac{0,01652}{v^3} + \frac{0,0012100}{v^4}$	82,5	82,7
200	$p = \frac{38,835}{v} - \frac{0,2385}{v^2} - \frac{0,01907}{v^3} + \frac{0,0009687}{v^4}$	94,2	94,0
250	$p = \frac{42,940}{v} - \frac{0,0230}{v^2} - \frac{0,02267}{v^3} + \frac{0,0007924}{v^4}$	105,5	105,5
300	$p = \frac{47,042}{v} - \frac{0,1546}{v^2} - \frac{0,02698}{v^3} + \frac{0,0006601}{v^4}$	116,7	116,8

Experimentally determined values of compressibility were smoothed in relation to pressures by entering the isometrics on the scale  $p - T$ . But this smoothing corrected the obtained results only to an insignificant degree. In all cases the points fit well on the  $p - T$  scale. Tables 2 and 3 show smoothed  $p - v - T$  values interpolated to whole numbers of temperature values for ammonia-hydrogen mixtures containing 43.75 and 57.49% ammonia.

Table 2. Values of  $p - v - T$  for a mixture of ammonia and hydrogen, containing 34.75% ammonia and 65.25% hydrogen

(1) Pressure in atmospheres

(2)  $v \text{ cm}^3/\text{mole}$

Давление, атм (1)				(2)
150° C	200° C	250° C	300° C	$v \text{ cm}^3/\text{mole}$
82,7	94,0	105,5	116,8	405,3
274,0	257,0	291,0	324,5	154,7
383,5	443,5	503,0	562,0	95,52
478,0	557,0	636,0	715,0	78,97
616,0	718,0	821,0	923,0	64,84
716,0	832,0	947,0	1063	58,28
860,0	1002	1144	1287	51,03

As seen from Tables 2 and 3, increases of the percent of hydrogen content of the mixture at the same pressures is accompanied by an increase of the molar volume of the mixture. This is, of course, explained by the lower compressibility of hydrogen in comparison with ammonia.

Table 4 shows  $p - v - T$  values for an ammonia-nitrogen mixture containing 44.40% ammonia (by volume), smoothed and interpolated to whole numbers of temperature values.

Comparison of the data from Tables 2 and 4 indicates that at equal pressures, despite the greater content of ammonia in its mixture with nitrogen, the mixture has a greater molar volume (temperature of 300°C, pressure of 1288 atmospheres) than the hydrogen-ammonia mixture. This is explained by the considerably lower compressibility of nitrogen at high temperatures and pressures in comparison with hydrogen.

Table 3. Values of  $p - v - T$  for an ammonia-hydrogen mixture, containing 57.49% ammonia and 42.51% hydrogen. (1) Pressure in atmospheres, (2)  $v$   $\text{cm}^3/\text{mole}$ , (3)  $p$  calculated by the Beattie-Bridgeman equation, (4) pressure, experimental.

Давление, атм ①				② $v$ $\text{cm}^3/\text{mole}$	$t^\circ \text{C}$	③ $p_{\text{вмч}}$ по уравнению Битти-Бриджмена, атм	④ $p_{\text{эксп}}$ , атм
150° C	200° C	250° C	300° C				
91,6 227,0	104,8 270,0	118,5 313,0	132,0 356,0	343,9 131,0	150	89,5	91,6
382,0 484,0 608,0	467,0 595,0 752,0	552,0 706,0 895,0	637,0 817,0 1038	78,42 64,31 54,42	200 250	104,3 118,5	104,8 118,5
892,0	1103	1314	1525	43,29	300	132,4	132,0

Table 4. Values of  $p - v - T$  for an ammonia-nitrogen mixture, containing 44.40% ammonia and 55.60% nitrogen.

Давление, атм ①				② $v$ $\text{cm}^3/\text{mole}$	$t^\circ \text{C}$	③ $p_{\text{вмч}}$ по уравнению Битти-Бриджмена, атм	④ $p_{\text{эксп}}$ , атм
150° C	200° C	250° C	300° C				
98,1 257,5 427,0 558,0 768,0 1330	113,1 306,0 518,0 684,0 942 1643	128,1 354,0 609,5 809,0 1115 —	143,1 402,7 700,8 935,0 1288 —	320,4 133,7 86,79 72,03 60,02 47,17	150 200 250 300	96,3 112,5 128,1 143,3	98,1 113,1 128,1 143,1

Table 5. Values of  $p - v - T$  for an ammonia-nitrogen-hydrogen mixture, containing 17.42% ammonia, 20.65% nitrogen and 61.95% hydrogen.

Давление, атм ①				② $v$ $\text{cm}^3/\text{mole}$	$t^\circ \text{C}$	③ $p_{\text{вмч}}$ по уравнению Битти-Бриджмена, атм	④ $p_{\text{эксп}}$ , атм
150° C	200° C	250° C	300° C				
72,5 208,0 360,0 455,0 577,0 803,0	81,5 232,0 409,0 518,0 577,0 916,0	90,2 256,0 458,0 581,0 739,0 1029	99,3 280,0 507,0 644,0 819 1141	485,4 181,8 110,3 91,03 76,01 60,18	150 200 250 300	71,8 81,1 90,2 99,2	72,5 81,5 90,2 99,3

Tables 5, 6 and 7 show smoothed values of  $p - v - T$  for ternary mixtures of nitrogen-hydrogen-ammonia containing 17.42%, 38% and 49.50% ammonia (by volume), interpolated to whole numbers of temperature values.

Table 6. Values of  $p - v - T$  for a mixture of ammonia-nitrogen-hydrogen, containing 38% ammonia, 15.5% nitrogen and 46.5% hydrogen.

Давление, атм ①				② $v$ см <sup>3</sup> /моль	$t^{\circ}C$	③ $p_{\text{выч}}$ по уравнению Битти-Бриджмена, атм	④ $p_{\text{выч}}$ атм
150° C	200° C	250° C	300° C				
88,5	100,0	111,7	123,5	380,0	150	86,6	88,5
425,0	495,0	565,0	635,0	86,32	200	99,3	100,0
539,0	632,0	725,0	818,0	126	250	111,7	111,7
696,0	816,0	936,0	1056	59,50	300	123,8	123,5
1007	1187	1367	1547	47,11			

Table 7. Values of  $p - v - T$  for a mixture of ammonia-nitrogen-hydrogen, containing 49.5% ammonia, 12.62% nitrogen and 37.88% hydrogen.

Давление, атм ①				② $v$ см <sup>3</sup> /моль	$t^{\circ}C$	③ $p_{\text{выч}}$ по уравнению Битти-Бриджмена, атм	④ $p_{\text{выч}}$ атм
150° C	200° C	250° C	300° C				
98,0	112,0	126,0	140,0	327,5	150	95,5	98,00
251,0	296,4	341,7	387,0	125,0	200	111,1	112,0
438,0	525,5	613,0	700,5	77,17	250	126,0	126,0
566,0	686,0	806,0	926,0	63,80	300	140,6	140,0
778,0	946,0	1114	1282	52,39			

It is interesting to note that the isometrics of the binary and ternary mixtures studied by us form straight lines for a broad region of temperatures and pressures, making a reliable extrapolation of this data possible for higher temperatures.

#### Error Evaluation.

The degree of accuracy of the obtained experimental data on the compressibility of nitrogen-hydrogen-ammonia mixtures depends on the precision of measurement of pressure, volume and temperature, on the purity of the components of the mixtures, and on the correctness of the Beattie-Bridgeman equation state which we used for the calculation of the molar volumes of the mixtures studied at pressures from 90 to 128 atmospheres and a temperature of 250°C. As indicated above, pressure

was measured with spring-type "model manometers" with a precision of 0.35%; the temperature fluctuation of  $\pm 0.15^{\circ}\text{C}$  can bring about a maximum pressure error of 0.08%. Divergencies in volume measurement, discovered by repeated piezometer calibrations, amounted to 0.05%. The 0.04% water content of the mixture cannot be a cause of noticeable error.

The error arising from use of the Beattie-Bridgeman equation, from data of Beattie and Beattie-Ikehara, and the congruence of  $p$  calculated and  $p$  experimental shown on Tables 1-7 (except at a temperature of  $150^{\circ}\text{C}$ ), apparently constitutes 0.5%. Thus, the total error of the obtained experimental data on the compressibility of nitrogen-hydrogen-ammonia mixtures may reach 1%.

#### Conclusions.

1. Compressibility of ammonia-hydrogen binary mixtures of two compositions, containing 34.75% and 57.49% ammonia was determined in the pressure range of 82.7 to 1525 atmospheres and at temperatures of 150, 200, 250 and  $300^{\circ}\text{C}$  with an accuracy of  $\pm 1.0\%$ .

2. Compressibility of the binary mixture ammonia-nitrogen, containing 44.40% ammonia and 56.60% nitrogen was determined in the pressure range of 98 to 1643 atmospheres and at temperatures of 150, 200, 250 and  $300^{\circ}\text{C}$  with an accuracy of  $\pm 1.0\%$ .

3. Compressibilities of three ternary mixtures of ammonia-nitrogen-hydrogen, with a nitrogen-hydrogen ratio of 1:3 and with an ammonia content of 17.42%, 38.0% and 49.50% were determined. Data was obtained for the pressure range from 72 to 1547 atmospheres at temperatures of 150, 200, 250 and  $300^{\circ}\text{C}$  with an accuracy of  $\pm 1.0\%$ .

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13. ABSTRACT		
<p>Data available in scientific literature on the compressibility of gas mixtures deals primarily with mixtures consisting of non-dipole gases. The mixtures of nitrogen-hydrogen, hydrogen-carbon dioxide, nitrogen-carbon dioxide, nitrogen-methane, hydrogen-methane, and methane-ethane have been investigated in a broad range of temperatures, pressures and compositions.</p> <p>Data on the compressibility of gas mixtures, one component of which is a dipole gas, however, is completely unsystematic, and is available only up to a pressure of 100 atmospheres.</p> <p>In our experiment we have determined the compressibility of binary ammonia-hydrogen and ammonia-nitrogen mixtures, as well as of ternary mixtures containing nitrogen, hydrogen and ammonia, for a number of compositions at high temperatures and pressures.</p> <p>The apparatus used was a modification of that used by Michels, who obtained exceptionally great precision.</p>		

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Compressibility Gas mixtures Ammonia/hydrogen mixtures Ammonia/nitrogen mixtures Nitrogen/hydrogen/ammonia mixtures High temperature High pressure Michel's method Michel's apparatus Beattie-Bridgeman equation Beattie-Ikehara data						

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